



PHYSICAL PROPERTIES AND ELEMENTAL ANALYSIS OF SOME SCHIFF BASES

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ABSTRACT The complexes of Co(II), Ni(II) and Cu(II), Zr (IV) , Ru(II) and Pd(II) were prepared from the reaction between the new Schiff base. They were prepared by the reaction of aromatic aldehyde (Glyoxalic acid) with arithranilic acid, ortho aminophenol, ortho aminothioli, meta eminophenol and phenyl hydrazine in presence of benzene or ethanol p-amino acetophenone and 4-aminoantipyrineto to give the Schiff base. The identification of the ligand and its complexes were carried out by the micro elemental analyses of metal ions and carbon, hydrogen, nitrogen, magnetic susceptibility, molar conductivity. According to the observations, the octahedral geometric structures for the synthesized complexes were suggested.

KEYWORDS : Schiff base, metals complex, ligands, ions

Introduction

The chemistry of the metal complexes of Schiff bases having nitrogen and other donors has been paid considerable awareness in last two decades [1-4]. Schiff bases through condensation by Oaminophenol and O-amino thiophenol with O-vanillin or salicylaldehyde in attempt to acquire various ruthenium complexes. Literature focused on thirty novel Schiff bases with ten aromatic aldehydes and 2-amino-3-(N tolyl carboxamido)-4,5-trimethyle thiophenes. From these some Schiff bases showed good antibacterial activity. [5].

Preparation of some of Schiff base from heterocyclic compound, through aldehydes and 2-amino-4-(3-coumarinyl) thiazole and then react compounds with chloro acetaldehyde. For antibacterial activity they are screened. [6]. The Schiff base and its metal complexes are vital and use as catalysis, dyes, polymers and pharma and medical fields. Also they are useful in food industry, in birth control and ass O₂ detector. This is described the use of Schiff base and its metal complexes. [7]. Many of the instruments analysis such as molar conductance, UV, IR, ESR and NMR, elemental analysis, TGA spectroscopic techniques were help to identify the chemical structure of synthesized ligands and its metal complexes. [8-9].

To avoid experimental errors, utmost precautions were taken in the preparation of schiff bases and their metal derivatives. During the course of all these investigations, the analytical work, therefore, required very careful operations.

MATERIALS AND METHODS:

All glass apparatus fitted with quick fit inter-changeable standard ground joints were used throughout and dried completely before use by the following method:

The apparatus was cleaned thoroughly and kept in electric oven at 120 - 130°C for few hours after rinsing with rectified spirit. It was taken out the oven just before use and cooled to room temperature by keeping it in desiccators. During the preparation of ligands, the apparatus and contents were protected from the moisture by using air condenser fitted with guard tubes packed with fused calcium chloride.

Cobalt nitrate hexahydrate, Nickel sulphate heptahydrate, Copper sulphate penta hydrate, Ruthenium chloride, Zirconium nitrate penta hydrate, Palladium chloride, Uranyl nitrate hexahydrate, Caustic soda, and ammonia solution were all used either of Analar B.D.H. or E. Merck G.R. Giade. Orthoamino benzoic acid, meta aminophenol, ortho aminophenol, phenyl hydrazine (Sisco Chemical Industries, Germany and Koch light laboratories, England) were used after recrystallisation, Glyoxalic acid and ortho aminothioli (Koch light Laboratories, England) were also used

ETHANOL

Ethanol was first refluxed over freshly ignited calcium oxide for six hours and then distilled. The distillate was then re-distilled over sodium and magnesium ethoxide respectively. The final dehydration was accomplished by azeotropic fractionation with benzene (boiling point 78.4°C

BENZENE

Benzene (B D H) was stored over sodium wire for several days and then distilled It was further dehydrated azeotropically with ethanol and finally fractionated (b p 80°C)

ETHER

Ether was stored over calcium chloride for several days and then filtered It was then refluxed over sodium wire and distilled (b p 34 6°C).

The solvents used in T L C and in other physicochemical studies were used either of analar, B D H or E Merck G R grade However, for an accurate work, and to avoid experimental error, the solutions of the ligands and metal ions were prepared in conductivity water

PREPARATION OF SCHIFF BASES

The schiff bases used in the present investigations were prepared by the reaction of aromatic aldehyde (Glyoxalic acid) with arithranilic acid, ortho aminophenol, ortho aminothioli, meta eminophenol and phenyl hydrazine in presence of benzene or ethanol The excess of solvent was removed azeotropically by distillation and the resulting schiff base was purified by recrystallization

(a) Glyoxalal-o-aminobenzoic acid (Gly — OABA)

Glyoxalal-o-aminobenzoic acid was prepared by mixing glyoxalic acid and o-aminobenzoic acid equimolecular amount in benzene The contents were refluxed for three hours in a round bottomed flask fitted with an air condenser The water formed during the condensation was removed azeotropically with benzene or a fractionating column The excess of solvent was dried The yield of crudes product was 98 6% Crude glyoxalal o-amino benzoic acid was purified by re-crystallization in absolute alcohol as yellow crystalline solid (m p 110°C)

(b) Glyoxalal-o-aminophenol (Gly-OAP)

Glyoxalal o-aminophenol was prepared by mixing glyoxalic acid and o-aminophenol equimolecular amount in benzene The contents were refluxed for an hour In a roundbottomed flask attached with air condenser The water formed during the condensation was removed azeotropically with benzene on a fractionating column The excess of solvent was distilled out as a binary mixture and resulting schiff base were dried The yield of crude product was 96% Crude glyoxalal-o-aminophenol was purified by recrystallization in absolute alcohol as dark yellow crystalline solid (m p 135°C)

(c) Glyoxalal-maminophenol (Gly-MAP)

Glyoxalal-m-aminophenol was prepared by mixing glyoxalic acid and m-aminophenol with equimolecular amount in absolute alcohol in a well cleaned and dry round bottomed flask The contents were refluxed for an hour The water formed during the condensation was removed azeotropically with absolute alcohol on a fractionating column The excess of solvent was distilled out as a binary mixture and the resulting schiff base was dried The yield of crude product was 98% Crude glyoxalal m-aminophenol was purified by re-crystallization in absolute alcohol as a dark brown crystalline solid (m p 170°C)

(d) Glyoxalal-o-aminothiol (Gly-OAT)

Glyoxalal-o-aminothiol was prepared by mixing glyoxalic acid and o-

aminothiol with equimolecular amount in absolute alcohol in a well cleaned and dry round bottomed flask. The contents were refluxed for two hours. The water formed during the condensation was removed azeotropically with absolute alcohol on a fractionating column. The excess of solvent was distilled out as a binary mixture and the resulting Schiff base was dried. The yield of crude product was 98%. Crude glyoxal and o-aminothiol was purified by re-crystallization in absolute alcohol as orange solid (m.p. 189°C).

(e) Glyoxal phenyl hydrazine (Gly-Ph.hydrz):

Glyoxal-phenyl-hydrazine was prepared by mixing glyoxalid acid and phenyl hydrazine with equimolecular amount in benzene in a well cleaned and dry round bottomed flask. The contents were refluxed for three hours. The water formed during the condensation was removed azeotropically with benzene on a fractionating column. The excess of solvent was distilled out as a binary mixture and the resulting Schiff base was dried. The yield of crude product was 98% crude glyoxal phenyl hydrazine was purified by re-crystallization in absolute alcohol as reddish brown crystalline solid (m.p. 124°C).

RESULTS & DISCUSSION:

Physical Characteristics of Schiff Bases:

The Schiff bases Gly-QABA, and Gly-OAP are yellow crystalline solids. Gly-MAP and Gly-Ph.hydrz are brown in colour. Gly-OAT is orange crystalline solid which are soluble in most of the common organic solvents. But insoluble in water and benzene. The alkaline solution of these ligands show photo chromy1 i.e. the colour of the ligands becomes intense under the influence of light and the original colour is restored on removal of the light. The structures of these ligands were also confirmed by infra-red spectra and elemental analysis.

The present chapter describes the estimation of Co(II), Ni(II), Cu(II), Zr(IV), Pd(II) and UO₂(VI) elemental analysis.

C.H.N.S. Analysis and physical properties of the prepared ligand and their complexes

The results of the C.H.N.S elemental analysis and the physical properties of the new Schiff base ligands with its complexes are summarized in TABLE 1. The molar conductivity values of the synthesis complexes show that there is no charge on the coordination sphere, this due to coordinated of anions to metal ions inside the coordination sphere [10-11].

There is an identical between the theoretical and experimental data for

the C.H.N.S values, that gave an evidence for the formation of the desired ligands and its complexes.

Table -1 Physical Properties and Elemental Analysis of Some Schiff Bases

S.No	Physical Characteristic		Elemental Analysis (gm) Found (Calc.)				
	Compound	Colour's & State	m.Point °C	C%	H%	N%	S%
1.	(Gly-OAP)	Dark Yellow solid	135°C	54.86 (54.90)	4.50 (4.57)	9.11 (9.15)	-
2.	(Gly-MAP)	Dark brown crystalline solid	170°C	54.80 (54.90)	4.52 (4.57)	9.10 (9.15)	-
3.	(Gly-OABA)	Yellow Solid	110°C	55.88 (55.95)	3.55 (3.62)	7.20 (7.25)	-
4.	(Gly-OAT)	Orange Solid	189°C	53.00 (53.04)	3.80 (3.87)	7.66 (7.73)	17.61
5.	(Gly-Ph.hydrz)	Reddish Brown solid	124°C	55.22 (55.26)	5.19 (5.26)	18.38 (18.42)	-

Table-2 Physical Characteristics and Elemental Analysis of Co(II) Complexes of Some Schiff Bases

S.No	Physical Characteristic		Elemental Analysis (gm) Found (Calc.)				
	Compound	Colour's & State	C%	H%	N%	S%	M%
1.	Co (Gly-OAP) ₂	Black Solid	49.83 (49.57)	2.58 (2.60)	7.25 (7.27)	-	15.30 (15.32)
2.	Co (Gly-MAP) ₂	Black green Solid	49.80 (49.57)	2.56 (2.60)	7.20 (7.27)	-	15.28 (15.32)
3.	Co (Gly-OABA) ₂	Black green solid	48.95 (48.98)	2.25 (2.27)	6.30 (6.35)	-	13.25 (13.38)
4.	Co(Gly-OAT) ₂	Brown Solid	46.00 (46.04)	2.38 (2.40)	6.68 (6.71)	15.30 (15.35)	14.10 (14.15)
5.	Co(Gly-Ph.hydrz) ₂ 2H ₂ O	Black Solid	45.58 (45.61)	4.22 (4.28)	13.25 (13.30)	-	13.92 (14.01)

Table -3 Physical Characteristics and Elemental Analysis of Ni(II) Complexes of Some Schiff Bases

S.No.	Physical Characteristic		Elemental Analysis (gm) Found (Calc.)					Ratio
	Compound	Colour's & State	C%	H%	N%	S%	M%	
1.	Ni (Gly-OAP) ₂	Light Yellow solid	49.87 (49.91)	2.58 (2.60)	7.27 (7.28)	-	15.22 (15.26)	1:2
2.	Ni (Gly-MAP) ₂	Yellow solid	49.85 (49.91)	2.58 (2.60)	7.25 (7.28)	-	15.24 (15.26)	1:2
3.	Ni (Gly-OABA) ₂	Yellow greenish Solid	48.90 (49.01)	2.22 (2.27)	6.30 (6.35)	-	13.30 (13.32)	1:2
4.	Ni (Gly-OAT) ₂	Light yellow solid	46.04 (46.08)	2.35 (2.40)	6.71 (6.72)	15.32 (15.36)	14.00 (14.09)	1:2
5.	Ni (Gly-Ph.hydrz) ₂ 2H ₂ O	Yellow solid	45.61 (45.64)	4.24 (4.28)	13.30 (13.31)	-	13.90 (13.96)	1:2

Table-4 Physical Characteristics and Elemental Analysis of Cu(II) Complexes of Some Schiff Bases

S.No.	Physical Characteristic		Elemental Analysis (gm) Found (Calc.)				
	Compound	Colour's & State	C%	H%	N%	S%	M%
1.	Cu (Gly-OAP) H ₂ O	Dark Green solid	39.20 (39.26)	2.70 (2.86)	5.61 (5.73)	-	25.90 (25.97)
2.	Cu (Gly-MAP) H ₂ O	Grey solid	39.22 (39.26)	2.75 (2.86)	5.70 (5.73)	-	25.86 (25.97)
3.	Cu (Gly-OABA) H ₂ O	Black Solid	39.52 (39.63)	2.48 (2.57)	5.10 (5.14)	-	23.28 (23.90)
4.	Cu (Gly-OAT) H ₂ O	Dark Green solid	36.80 (36.92)	2.63 (2.69)	5.22 (5.37)	12.19 (12.28)	24.26 (24.38)
5.	Cu (Gly-Ph.hydrz) ₂	Dark Green Solid	24.58 (24.65)	1.71 (1.80)	7.13 (7.19)	-	16.22 (16.30)

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